

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Donald B. Appleby et al : Group Art Unit: 1211
Serial No.: 08/360,184 : Examiner: E. White
Filed: December 20, 1994 :
For: **Polyol Polyester Synthesis**

DECLARATION UNDER 37 C.F.R. §1.608 OF PATRICK J. CORRIGAN

Assistant Commissioner for Patents
Washington, DC 20231

Dear Sir:

I, PATRICK J. CORRIGAN, declare that:

1. I am a co-inventor of and am familiar with the present Appleby et al U.S. patent application.
2. I received a Bachelor of Science Degree in Chemistry from The United States Naval Academy in 1975 and a Masters of Science Degree in Chemical Engineering from the University of Notre Dame in 1982.
3. I have been employed by the assignee of the present application, The Procter & Gamble Company, from about June 1982 to the present time. During my employment with The Procter & Gamble Company, I have worked, inter alia, on developing a process for the synthesis of

polyol fatty acid polyesters, specifically sucrose fatty acid polyesters, referred to herein as sucrose polyesters, by reaction of sucrose and fatty acid methyl esters.

4. During my employment with The Procter & Gamble Company, I have, from time-to-time, prepared and distributed biweekly and monthly reports accurately describing my activities, and/or activities of those under my direction and control, during the prior two week or monthly period which was the subject of the report. For each report, I accurately set forth therein the ending date for the prior two week or monthly period which was the subject of the report.

5. I have reviewed the respective Declarations Under 37 C.F.R. § 1.608 of Roger S. Berger and Ephraim L. Kelly and Exhibits Nos. 28-38 identified in those Declarations, all of which Declarations and Exhibits are submitted herewith. The Berger and Kelly Declarations accurately describe experiments conducted by them under my direction and control for studying and developing the sucrose polyester process and accurately describe the procedures for recording the general nature and results of such experiments as respectively described therein.

6. Under my direction and control, a three liter, five pound per hour (5 pph) laboratory scale continuous reactor system for polyol fatty acid polyester production was constructed between January 1989 and July 1989. I have reviewed Exhibits 39 and 40 and confirm that they are accurate copies of, respectively, my Industrial Chemicals Product Development Biweekly Report for the period ending January 25, 1989 and my Chemicals Product Development Division Biweekly Report for the period ending July 26, 1989, and that I accurately described therein my activities for the biweekly periods ending January 25, 1989 and July 26, 1989, respectively. As set forth in Exhibit

39, the construction of the laboratory scale continuous reaction system began on or about the biweekly period ending January 25, 1989 and the purpose of this reactor system was to bridge the gap between one liter batch reactors and the larger pilot plant continuous systems and to permit, inter alia, fundamental studies of the FG (sucrose-fatty acid methyl ester) reaction and rapid assessment of continuous system parameter changes. As set forth in Exhibit 40, the laboratory continuous reaction system became operational in July 1989. The laboratory continuous reaction system was subsequently used to investigate various parameters for conducting the sucrose polyester process on a continuous basis, which parameters are disclosed throughout the present Appleby et al application.

7. I have reviewed Exhibit 41 and confirm that it is an accurate copy of my Industrial Chemicals Product Development Biweekly Report for the period ending May 17, 1989 and that I accurately described therein my activities for the biweekly period ending May 17, 1989. As set forth in Exhibit 41, studies under my direction and control were conducted in May 1989 to investigate the amount of soap emulsifier which was required in the sucrose polyester reaction. As accurately described in the Kelly Declaration, ¶¶9-12, and Exhibit 36, a matrix of experiments was performed by Mr. Kelly under my direction and control in May 1989 to evaluate the effect of various soap levels on the sucrose polyester reaction. Soap to polyol molar ratios of 0.34:1, 0.42:1, and 0.46:1 (Kelly Declaration, ¶¶11, 12 and 10, respectively) were employed. These ratios are within the ranges disclosed as desirable at page 15, lines 15-19 of the Appleby et al application. Sucrose polyester product comprising 97% or greater octaester was obtained using these low soap levels, thereby demonstrating that low levels of soap could be used in the sucrose polyester reaction, as disclosed in the Appleby et al application at page 15, lines 4-10.

8. I have reviewed Exhibits 42 and 43 and confirm that they are accurate copies of my Chemical Product Development Division Biweekly Reports for the periods ending August 23 and September 6, 1989, and that I accurately described therein my activities for the biweekly periods ending August 23 and September 6, 1989, respectively. As set forth in Exhibits 42 and 43, studies under my direction and control were conducted in August and September of 1989 to investigate the size of the sucrose which was used in the sucrose polyester reaction. As set forth in Exhibit 42, these studies were intended to determine the effect of sucrose particle size distributions on the continuous reaction as well as to determine the lower size limit, if any, for sucrose particles, and the laboratory continuous reactor system was ideally suited for these tests because it required only a small amount of sucrose (e.g., 2 kg) such that the sucrose could be easily sieved to obtain the desired size distributions. As set forth in Exhibit 43, fluid energy (jet) milling of sucrose was to be investigated as a means for producing sucrose particle sizes less than 10 microns.

9. As accurately described in the Berger Declaration, ¶¶6-8, and Exhibit 28, a matrix of experiments was performed by Mr. Berger under my direction and control in September 1989 to investigate the effect of various sucrose particle sizes on the sucrose polyester reaction using the laboratory continuous reactor system. Sucrose particle sizes less than about 63 microns and less than about 38 microns were tested (Berger Declaration, ¶¶7 and 8, respectively), with the smaller sucrose particles achieving the higher average degree of esterification based on mole fractions, i.e., an I-bar of 2.97 obtained with the less than about 38 micron sucrose particles versus an I-bar of 2.77 obtained with the less than about 63 micron sucrose particles, and the greater sucrose utilization, i.e., 3% unreacted sucrose at 4.5 hours using less than about 38 micron sucrose particles versus 5% unreacted sucrose at 4.5 hours using the less than about 63 micron sucrose particles. These results

demonstrated faster and more efficient reactions were obtained using smaller polyol particles, as set forth in the Appleby et al application at page 13, line 6 - page 14, line 4.

10. As accurately described in the Berger Declaration, ¶¶11-14, and Exhibit 30, a matrix of experiments was performed by Mr. Berger under my direction and control in December 1989 to investigate the effect of jet milled sucrose particles on the sucrose polyester reaction using the laboratory continuous reactor system. In December 1989, a jet mill was obtained and reactions using jet-milled sucrose having particles sizes of about 10 microns or less were performed in the continuous laboratory reactor system (Berger Declaration, ¶¶12-14). The sucrose particles sizes employed in these experiments were within the preferred ranges disclosed in the Appleby et al application at pages 14, lines 1-4. These tests resulted in sucrose polyester products having equivalent or increased average degrees of esterification (\bar{I} -bar values of about 3.20, 2.86 and 2.84 at 4.5 hrs, Exhibit 30) and lower amounts of unreacted sucrose (about 0.63%, 1.13% and 0.71% at 4.5 hrs, Exhibit 30) than the tests conducted in September 1989 and previously discussed in paragraph 9 herein (\bar{I} -bar values of about 2.77 and 2.97 and 5.11% and 3.78% unreacted sucrose at 4.5 hrs, Exhibit 28) which used larger sized sucrose particles. These results further demonstrate that small sucrose particle sizes increase sucrose polyester reaction speed and efficiency as disclosed at page 13, lines 8-10 of the Appleby et al application.

11. As accurately described in the Kelly Declaration, ¶¶15-20, and Exhibit 38, a matrix of experiments was performed by Mr. Kelly under my direction and control in September and October of 1989 to investigate various fatty acid methyl ester to polyol molar ratios in the sucrose polyester reaction. Molar ratios of total ester reactant to esterifiable sites on the polyol of about 1:1

and about 1.16:1 were tested (Kelly Declaration, ¶¶ 16 and 18-20 (1:1 molar ratio) and ¶17 (1.16:1 molar ratio)). These molar ratios are within the ranges disclosed at page 17, lines 8-11 of the Appleby et al application. The reactions provided sucrose polyester product comprising 74% or greater octaester and I-bar values of at least 7.65, and therefore demonstrated that low ester to polyol ratios can be used in the sucrose polyester reaction, as disclosed in the Appleby et al application at page 17, lines 7-37.

12. As accurately described in the Berger Declaration, ¶¶15-21, and Exhibit 31, a matrix of experiments was performed by Mr. Berger under my direction and control in February and March of 1990 to investigate the use of higher later stage reaction pressures in the sucrose polyester reaction as disclosed at page 18, lines 16-36 of the Appleby et al application. The experiments demonstrated that sucrose polyester comprising about 75% or greater octaester could be achieved using later stage reaction pressures of 15 and 25 mm Hg and nitrogen sparging (Berger Declaration, ¶¶16-19), and that sucrose polyester comprising greater than about 70% octaester could be achieved using later stage reaction pressures of 30 mm Hg and 45 mm Hg and nitrogen sparging (Berger Declaration, ¶¶20 and 21).

13. I have reviewed Exhibit 44 and confirm that it is an accurate copy of my Chemicals Product Development Division - Olestra Process Monthly Report for the period ending March 1, 1990 and that I accurately described therein my activities for the monthly period ending March 1, 1990. As set forth in Exhibit 44, the standard sucrose polyester reaction employing a later stage pressure of 0.5-1 mm Hg resulted in a reaction product comprising greater than 1000 ppm difatty ketone (DFK). The experiments described in paragraph 12 herein also investigated the effect of the

later stage reaction pressure on the amount of DFK in the reaction product. As further set forth in Exhibit 31, difatty ketone levels of about 300 ppm or less were achieved with second stage pressures of 15, 25, 30 and 45 mm Hg (Berger Declaration, ¶¶16-21). This level of DFK is within the range disclosed at page 24, line 13-15 of the Appleby et al application.

14. As accurately described in the Berger Declaration, ¶¶22-29, and Exhibit 32, another matrix of experiments was performed by Mr. Berger under my direction and control in February and May of 1990 to investigate the use of alternate catalysts, such as sodium methoxide and potassium methoxide, in the sucrose polyester reaction. These alternate catalysts were added in a later stage of the reaction and produced sucrose polyester products having I-bar values of greater than about 7 (Berger Declaration, ¶¶23-29). These experiments therefore demonstrated the feasibility of using the alternate catalysts and their addition in a later stage of the reaction, as disclosed at page 14, lines 27-32 of the Appleby et al application.

15. I have reviewed Exhibit 45 and confirm that it is an accurate copy of a Development Record which I prepared and submitted to the Patent Division of The Procter & Gamble Company on or about March 14, 1990. As indicated at the bottom of page 1 of Exhibit 45, this Development Record was assigned Development Record No. 7988. The Development Record is directed to improvements in the sucrose polyester reaction wherein low ester to sucrose molar ratios and small particle size sucrose are employed. The subject matter of the Development Record of Exhibit 45 was incorporated into the Appleby et al application, for example, at page 17, lines 7-37.

16. As accurately described in the Berger Declaration, ¶¶30-31, and Exhibit 33, another experiment was performed by Mr. Berger under my direction and control in March 1990 to investigate, inter alia, the effect of lower temperatures and higher pressures on the formation of difatty ketones in later stages of a sucrose polyester reaction. During a later stage of the reaction, the reaction temperature was reduced from 135°C to 100°C and the reaction pressure was increased from 15 mm Hg to 30 mm Hg for the remainder of the reaction. The sucrose polyester product comprised about 89.3% octaester, and had an average degree of esterification of about 7.80 and a difatty ketone level of 201 ppm, all at 14.5 hours, whereby the experiment demonstrated the feasibility of producing sucrose polyester product with low DFK content and high octaester content using lower temperatures and higher pressures as set forth in the Appleby et al application at page 23, lines 7-14.

17. As accurately described in the Berger Declaration, ¶¶32-34, and Exhibit 34, another matrix of experiments was performed by Mr. Berger under my direction and control in May 1990 to investigate, inter alia, the effect of various fatty acid methyl ester to sucrose molar ratios on the sucrose polyester reaction. A molar ratio of fatty acid methyl ester to esterifiable sites on the polyol of about 1.3:1 was employed while using powdered or granular sucrose and potassium carbonate catalyst in respective experiments. The sucrose polyester products comprised more than about 85% octaester and had an average degree of esterification of about 7.8 at 6.5 hours, thereby further demonstrating that low molar ratios of fatty acid methyl ester to esterifiable sites on the polyol as set forth in the Appleby et al application at page 17, lines 8-10 were suitable.

18. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

By: Patrick J. Corrigan
PATRICK J. CORRIGAN

Date: 3/29/99

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